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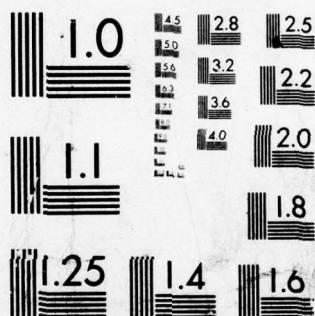
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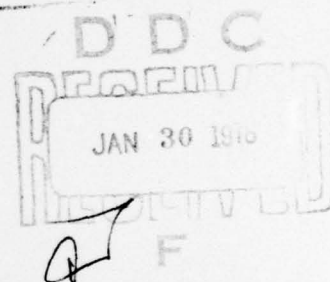
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**ORIGIN AND CONTROL OF RESIDUAL  
STRESSES IN AUTOMATICALLY DEPOSITED MATERIALS**

**FINAL REPORT  
DCI F-137**

**RICHARD E. ENGDAHL  
NOVEMBER 29, 1977**



**U.S. ARMY RESEARCH OFFICE**

**DAAG29-76-C-0030**

**DEPOSITS & COMPOSITES INCORPORATED  
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| For the first portion of the program, "The Origin and<br>Control of Residual Stress in Atomically Deposited Materials",<br>the emphasis was placed on identifying the origin of residual<br>stress in the model material CVD-SiC.<br>The initial assumption was that the origin of the residual<br>stress could be correlated with growth morphology. Parameters<br>such as temperature, hydrogen/silane ratios, flow rates, |                       |  |  |

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pressure and reactant species were varied in an attempt to reveal the stress/morphology relationship.

No apparent pattern emerged based on the initial assumption, but a relationship did become evident between extraneous gases and residual stress. This result required that the study program be recast along new lines that would emphasize measurement of the influence of additions of gases such as nitrogen and oxygen.

Equipment was designed and set up to produce 1<sup>mm</sup> diameter sample tubes of CVD-SiC. The arrangement permitted additions of various extraneous gases to the deposition species at concentrations that would exaggerate their influence on residual stress. This revised program has been proposed to be conducted during the next phase of the work.

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# ORIGIN AND CONTROL OF RESIDUAL STRESS IN ATOMICALLY DEPOSITED MATERIALS

## 1. INTRODUCTION

The requirements for improved energy conversion systems have created a need for high temperature (room temperature to at least 1400° C.) structural ceramics that demonstrate reliable high strength, exhibit low oxidation rates and have essentially zero creep. To accomplish this task, a family of superceramics is under development. Candidates have been selected and extensive work is underway for utilizing relatively conventional ceramic processing techniques, such as, hot pressing and/or sintering. A small effort is underway for the less conventional chemical vapor deposition processes. One of the prime CVD candidates is CVD silicon carbide (SiC), which has been made in ultra pure form and produced with theoretical density. In addition, the material has demonstrated the highest strength, lowest creep rate and lowest oxidation rates at temperatures to 1500° C. of any ceramic currently under evaluation. Further, the material has been deposited in the complex forms required for heat exchangers and gas turbine parts (1) of the type required to advance the state-of-the-art of energy conversion systems.

An inhibiting problem with atomically deposited materials has been residual stress. The strains and resulting stresses may arise from various anisotropic factors (2) and from terms, such as the thermal expansion mismatch at the substrate surface. However, not all of the observed strains arise from these basic physical factors, rather, they appear to be the result of the deposition parameters, various deposition additions or the result of specific growth patterns.

Residual stress is a phenomenon generally observed in atomically deposited material by processes as different as electroplating, chemical vapor deposition, and ion plating. The produced deposits may vary from highly stressed to essentially stress-free, under what has been believed to be similar or only slightly different conditions. In the past, most of the exploratory work on stresses in atomically deposited material has been related to electrolytically deposited material (3). These studies have demonstrated that small differences in the plating baths and circumstances can yield large differences in the residual stress of the produced material. However, to date no coordinated theory for internal stress for electrolytically deposited material has been formulated.



Stresses in material such as pyrolytic graphite have been studied. For this material, strains induced by the anisotropic properties are believed to be the prime factors contributing to the stress problem. Thus, these studies have not concentrated on the influence of the deposition parameters, but rather on the basic material properties.

This program has been directed at identifying the chemical vapor deposition parameters that appear to influence the residual stress in silicon carbide. It is believed that once the specific terms have been identified with adequate precision so that residual stress can be increased or decreased at will, then the material so produced will reveal significant information pertinent to the origin of residual stress.

## 2. SUMMARY OF RESULTS

The program to seek out the origin and control of residual stress was organized on the basis that residual stresses are the result of the material morphology. Thus, it was expected that if the material could be grown under various temperatures, flow rates, reactant ratios and reactant species, the deposits would reveal a pattern that would indicate the origin of residual stress. With this information control procedures could be established and a relevant theory postulated.

The program produced material at various temperatures with various reactant species and with various hydrogen to reactant ratio with various flow velocities. Studies of the produced material indicated that the residual stress cracking and grown in cracks were random in nature and could not be clearly associated with the growth morphology. This is not to say that growth morphology has no effect on the intrinsic and/or residual strength values, but that other factors play a more dominant role in the determination of residual stress.

During the course of the examinations, it was noted that one parameter did correlate with the observed residual stress. This factor was the condition of the vacuum pump. Since the effluent products from chemical vapor deposition are very corrosive to the process vacuum pumps, repair work is constantly required to maintain the shaft seals, bearing and check valves. It was observed toward the end of the first year that regardless of the deposition parameters the residual stresses were lower for the first runs after the pump was repaired. To further confirm the importance of this factor a new vacuum pump was purchased and used to produce an additional series of runs. These runs confirmed that reduced residual stress levels were associated with improved pump conditions.

Residual stress values are difficult to measure directly in thick, high modulus material and, therefore, we have resorted to sectioning the material and examining the surface for cracks that might originate at the first deposited layer, or at internal locations where the cracks have been grown over. Both crack types have been observed and the extent, frequency, and location of the cracks used as an indicator of the magnitude and sign of the residual stress.

With the information on the influence of extraneous gases on residual stress, the program plan was revised to concentrate on determining the gas specie or species and concentration associated with the various residual stress values. To make this measurement and correlation a residual gas analyzer was set up so that selected individual gases could be added to the deposition gases in varying amounts. The work to accomplish this portion of the plan is scheduled for the second year effort.



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